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Characterization of Particulate Emissions from In-Use Diesel Vehicles

New York State Dept. of Environmental Conservation, Albany

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Research Triangle Park, NC

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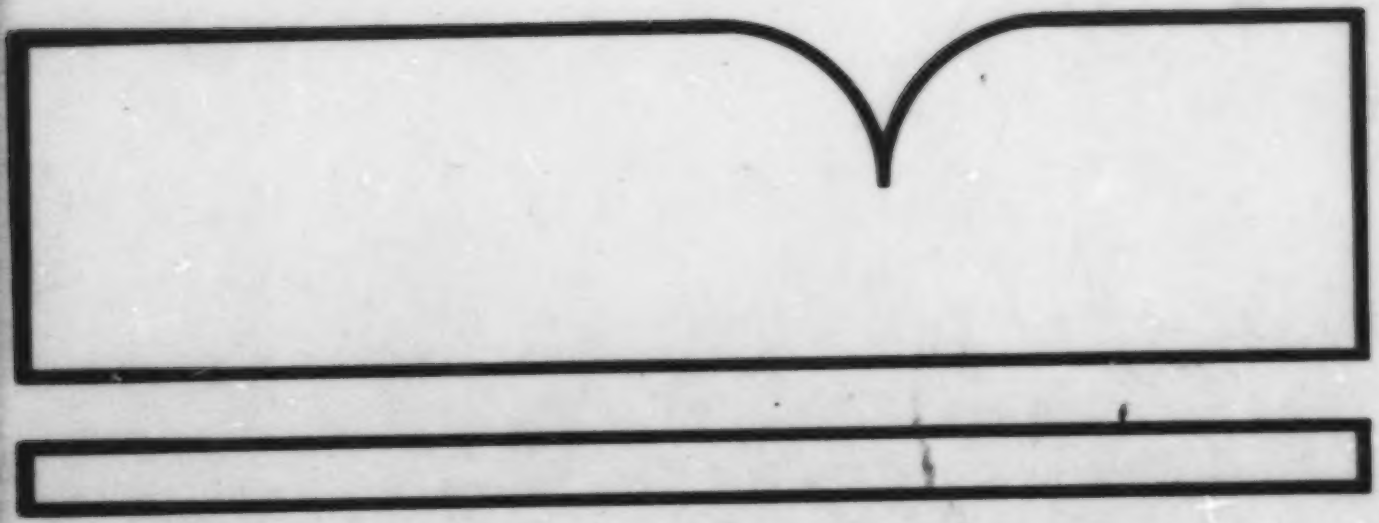

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INTRODUCTION

Background of the Problem - The importance of resource conservation is becoming generally felt as a relentless fact, and this presents new opportunities for fuel efficient combustion technology, such as the diesel engine. The number of diesel powered vehicles in all weight classes is thus expected to rise based on the inherent fuel consumption advantages of the diesel relative to comparable spark-ignition engines. However, the time scale for implementing major changes in combustion technology is large, even when measured in years, and the characteristic particulate emission of the diesel dictate the present need to carefully evaluate the impact of a trend toward diesel combustion. The chemical complexity of diesel particulate has impeded progress in even identifying important considerations, while biological screening assays, such as the Ames test, have increased awareness that some of the myriad of compounds carried by the particulate should be prudently considered as possible carcinogens. The lack of a definitive chemical basis for understanding the observed bioassay indication hinders constructive interaction among various areas impacting the overall issue, such as health effects, atmospheric chemistry, control technology options and effectiveness, regulatory action, and especially the underlying need for sampling and measurement methodologies for the bio-active compounds themselves.

Previous Experimental Work - The physical and chemical characterization and sampling and measurement methodologies for diesel emissions have been extensively reported for various engines, fuels and test conditions (1-15)*. The use of air dilution of vehicle exhaust followed by collection of particulate samples by filtration has been examined for application to diesel particulate by various investigations (16-20) and is the specified method for particulate measurement certification tests (21). Little chemical characterization data is presently available from in-use light-duty diesel engines, although gaseous, particulate and fuel economy results from two groups of General Motors 5.7 liter diesels have been recently reported (22,23).

The soluble organic fraction (SOF) of diesel particulate has been examined by the Ames bioassay

* Numbers in parentheses designate References at end of paper.

ABSTRACT

Particulate emissions data are presented from a group of 19 in-use diesel automobiles. Five driving cycles and three fuel/lubricating oil combinations have been used to obtain particulate emissions data and also to collect particulate samples for chemical and bioassay characterization studies. Particulate samples are solvent extracted with dichloromethane to obtain the soluble organic fraction (SOF) of the emitted particulate. Particulate and SOF data are presented for emission/distance, emission/time and emission/fuel consumed basis by vehicle manufacture, driving cycle, and fuel/oil subcategory. Some SOF samples have been used in the bioassay test of Ames, and these results are presented for different vehicles and driving cycles. Particulate emissions, SOF emissions and bioassay activity results are presented in terms of mileage accumulation effects.

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screening test and found to contain mutagenic substances (24-26). At a recent EPA conference on diesel emissions (27) several authors contributed in this area. Presently little is known of the chemical identity of the mutagens; and evidence has been reported (28) demonstrating an increase in mutagenic response with increased NO_2 levels in the exhaust dilution apparatus and the formation mutagen artifacts in particulate samples from systems to which NO_2 has been added. It is therefore difficult to assess the effects of sampling conditions on mutagenic response. Sampling procedures valid for emissions measurement cannot be extrapolated to be quantitative sampling procedures for bioactive compounds, even though nothing better may be available until further work in this area is performed.

General Plan of the New York State Study - The study underway at New York State recognizes the need for emission factors from in-use diesel vehicles. Particulate emissions samples are quantitatively collected, solvent extracted and bioassayed to determine the effects of vehicle design, fuels, oils and test conditions. Emissions and fuel consumption data are being collected from a sample of in-use diesel cars, which currently number 19 vehicles. A test protocol has been adopted to establish the sample group that involves about one week of dynamometer testing to obtain gaseous and particulate emissions data, and includes collection of particulate samples of sufficient size from which to perform chemical characterization and bio-assay tests. Sample group vehicles are re-tested to accumulate information on how vehicle ageing changes measured parameters. The vehicle test protocol adopted includes five vehicle operating modes (driving cycles) repeated with three combinations of engine fuel and lubricating oil. The fuel/oil combinations include testing the vehicle with 1) "as received" fuel and oil, 2) project reference fuel and "as received" oil, 3) project reference fuel and fresh lubricating oil. Particulate samples are collected by a standard dilution tunnel technique, followed by solvent extraction with dichloromethane to obtain the soluble organic fraction (SOF), or particulate extract.

A parallel effort to the in-use vehicle testing is directed toward developing SOF chemical characterization data to identify the mutagenic compounds present. The chemical complexity of the SOF, and the fact that the mutagenic activity is not attributable to the major constituents, makes identification of the mutagens difficult, and thus detailed chemical studies in this project are presently limited to a few select samples. To initiate chemical studies, three vehicles (Oldsmobile Cutlass wagon 5.7 liter V8, VW Rabbit 4-speed, and Mercedes-Benz 300D automatic) were operated on project reference fuel for approximately 30 days to collect large samples of particulate (50-100 g) for extraction, continuing chemical characterization and bioassay. Progress on these chemical studies has been reported elsewhere, (29-34) and is not included in this paper.

The in-use vehicle emissions fuel economy test results, chemical characterization of large particulate samples, and bioassay results from both in-use vehicle samples and the large samples are major complementary elements of the overall project.

VEHICLE EXPERIMENTAL PROCEDURES

Vehicle Testing Equipment - The major thrust of particulate characterization has been the collection of particulate samples according to 1981 EPA procedures (21) for new vehicle certification using a standard Constant Volume Sampler (CVS) air dilution of the vehicle exhaust followed by particulate sampling in a dilution tunnel. To obtain samples of sufficient size for subsequent chemical and bioassay analyses from discrete vehicle tests, a scaled-up particulate sampling system was added to the dilution tunnel. Fig. 1 is a schematic of

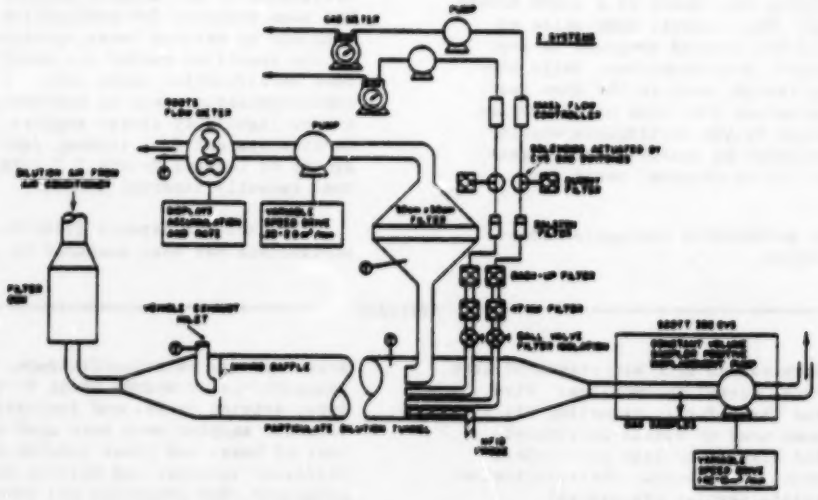

Fig. 1-Schematic of Particulate Collection Apparatus.

the exhaust dilution and particulate sampling equipment. Particulate mass emissions for vehicle tests are determined by proportional sampling of dilute exhaust by either of the two 47 mm filter systems. Each filter can be isolated from the probe and dilution tunnel by smooth-bore ball valves to permit changing of clean and loaded filters during vehicle testing. Each 47 mm filter is equipped with an identical 47 mm backup filter immediately downstream of the primary filter. Constant sampling flow is maintained as particulate is progressively deposited on the filter by means of a mass flow controller (Tylan Model FC-202) capable of maintaining constant mass flow over a wide range of gas temperatures and pressures. These flow controllers operate from a common power supply and display panel, and have individually adjustable mass flow rate set-points. These probe systems are flow calibrated each week with standard volumetric gas meters.

The particulate sampling system for obtaining bulk soot samples for extraction uses a filter assembly developed by EPA employing a 50 cm x 50 cm (20 in x 20 in) square filter mat. A nominal flow of 2.8 m^3/min (100 cfm) can be drawn through this filter and this provides 0.5 to 2 g of particulate from typical vehicle tests. Although not used for quantitative measurement of total particulate mass emissions, this system must include accurate flow measurement since the volumes filtered are a significant contribution to the total dilute exhaust volume, and this sampler is used during vehicle tests including other gaseous and particulate emissions determinations. The dilute exhaust volume passing through the 50 cm x 50 cm filter is light measured by a Roots-type totalizing flow meter (Dresser Industries model 5M125) mounted on the vent side of the blower used to draw sample through the filter (Fig. 1).
Continuously recorded temperature measurement at the Roots meter exit is used to correct the volumetric data to standard conditions. Propane tests with both CVS and 50 cm x 50 cm operating verify the flow measurement calibration of the complete system.

The filter media used in both the 47 mm and 50 cm x 50 cm filter systems is Pallflex Products Corporation type T60A20 teflon coated glass fiber. The nominal flows of the 47 mm and 50 cm x 50 cm system are 9 l/min and 2.26 x 10^3 l/min respectively, referenced to 0° C. Dilute exhaust flow per area of filter exposure is 0.014 l/sec/cm^2 and 0.018 l/sec/cm^2 for the 47 mm and 50 cm x 50 cm filters respectively, both within the EPA specified range for particulate measurement (.00951 < flow < .0272 l/sec/cm^2 at 0°C). Operation of the 50 cm x 50 cm filter in conjunction with the constant volume sampler (CVS) requires the 50 cm x 50 cm system to be started before the CVS to avoid filter tearing.

The volume filtered through the 50 cm x 50 cm system during emissions testing is obtained from a digital accumulator on the Roots meter actuated in syncronization with other sampling devices by the CVS bag fill switches.

The maximum allowable temperature of dilute exhaust for particulate sampling by EPA procedure is 52°C, and this requirement directly affects the dilution air volume requirements necessary to cool the vehicle exhaust prior to sampling. Sampling requirements for the variety of vehicles, engine sizes, inertia weights, and vehicle operating modes encountered in this study are best met with a range of dilution air flows. The need to obtain measurable levels of gaseous species simultaneous with particulate sampling prohibits the dilution air flow from being set at one high value to satisfy only the particulate sampling requirements. To accommodate these needs, an existing positive-displacement-pump (PDP) CVS was modified by addition of a 7.50 kw variable frequency 3Ø motor controller to give a variable speed motor drive providing continuous adjustment of CVS flow between 1.5-15 m^3/min (50-525 cfm). In combination with the 2.8 m^3/min (100 cfm) bulk particulate sampler, and the conditioned-air inlet to the dilution air filter box, the range of CVS flows available has satisfied the 52°C maximum temperature requirement for all vehicle test conditions. CVS + 50 cm x 50 cm filter flows have been consistently set to give a total dilute exhaust flow of 11.4 m^3/min (400 cfm) and 17 m^3/min (600 cfm) for Volkswagon and General Motors vehicles respectively. Strip chart recordings of dilute exhaust and Roots meter temperature are obtained for all tests. The PDP CVS blower is mass flow calibrated as a function of blower speed, and 132 daily propane tests since the start of in-use vehicle testing have averaged 0.75%$\pm$ 1.8% error.

Connection between the vehicle tailpipe and the dilution tunnel is either by a short length (approximately 1 m) of 7.6 cm (3 in) uninsulated flexible exhaust tube for vehicles with tailpipe exit near the tunnel (passenger side) or by a longer connector to vehicles with a tailpipe connector on the side of the vehicle opposite the tunnel location (driver side). Two types of connector have been utilized in this latter case. Through the first 17 vehicle tests, this connector was of the same type as above, but of sufficient length (4 m) to reach the tailpipe. For subsequent vehicle tests a smooth stainless steel tube (7.6 cm x 3 m) lagged with 5 cm of fiberglass insulation was used to link the tunnel to the tailpipe with short flexible uninsulated sections to make the connections. Particulate emission tests with the insulated and uninsulated systems showed approximately a 5% increase for the insulated case. EPA (35) has reported on the effect of tailpipe hook-up on observed particulate emissions. After nine months of daily testing the dilution tunnel was carefully cleaned. From the weight of tunnel scrapings and an estimate of the total mass of particulate entering the tunnel in the course of testing, the particulate losses in the tunnel are estimated to be 3%.

Gaseous emissions and fuel consumption are measured by the 1979 FTP simultaneously with particulate sampling by the 1981 FTP. The emissions chassis dynamometer used is a Clayton CTE-50, equipped with automatic road-load and direct-drive variable inertia (250 lb inertia increments to 3,000 lb, 500 lb inertia increments above 3,000 lb) The vehicle test bay is maintained at constant temperature by two air conditioning/heating/humidification/dehumidification units with a combined capacity of 106 kw and circulating 425 m^3/min conditioned air onto the vehicle positioned on the dynamometer. In order not to abuse privately owned vehicle tires on the dynamometer, and to aid in uniformity of test conditions, all testing was performed on duplicate tires, identical to

vehicle manufacturer specifications, inflated to 310 KPa (45 psig).

Test Cycle Sequence - Vehicles included in the sample group are brought to the Department of Environmental Conservation's Automotive Emissions Laboratory (AEL) in Latham, New York for a week-long comprehensive battery of tests to complete one "vehicle-test." Thirty-four such vehicle tests on nineteen vehicles constitute the data base of this paper. Each vehicle test is broken down into three repetitive "test-phases" according to the fuel and lubricating oil combination used. The three combinations of fuel and lubricating oil corresponding to each test phase are: Phase I - vehicle tested in as-received condition, Phase II - vehicle tested with fuel stored at AEL as a project reference fuel, and Phase III - vehicle tested with reference fuel after crankcase oil and oil filter are replaced with new oil and filter of manufacturer specification.

Five different vehicle driving schedules were used to obtain data for different vehicle operating modes. (Reference (5) gives speed-time traces and general cycle characteristics) The test cycles used were: 1979 Federal Test Procedure (FTP), Congested Freeway Driving Schedule (CFDS), Highway Fuel Economy Test (HFET), 50 mph cruise (50C) and idle.

During the three segments of the FTP individual 47 mm diameter particulate samples were obtained for each bag; however, a single composite bulk particulate sample was collected on the 30 cm x 50 cm filter during all three bags. CFDS test segments included particulate collection on both 47mm probes to verify system balance and operation. The HFET cycles were always run in blocks of three back-to-back cycles separated by 30 s idle periods. A single bulk sample was always collected from all three HFET cycles in any one block, but only during the middle HFET were the 47 mm filter samples and gaseous measurements performed.

Table 1-Sequence For One Complete Vehicle Test

Test Segment	Particulate Filter	Gaseous Analyzer	Test Phase	Bag
Procure Vehicle				
Prepare for Testing				
30 min @ 30 mph pre-test conditioning			I - as delivered fuel and oil	one
30 min @ 50 mph	Yes			
CFDS	Yes			
HFET x 3	Yes			
Overnight soak				
FTP	Yes	Yes		
CFDS	Yes	Yes		
HFET x 3	Yes	Yes		
30 min @ idle	Yes			
Change Fuel System to AEL Control Fuel				two
30 min @ 30 mph pre-test conditioning			II - control fuel, as delivered oil	
30 min @ 50 mph	Yes			
CFDS	Yes			
HFET x 3	Yes			
Overnight soak				
FTP	Yes	Yes		
CFDS	Yes	Yes		
HFET x 3	Yes	Yes		
30 min @ idle	Yes			
Change Oil to Fresh Oil of Manufacturers Recommendation				three
30 min @ 50 mph cruise pre-test conditioning			III - control fuel, fresh oil	
30 min @ 50 mph cruise	Yes			
CFDS	Yes			
HFET x 3	Yes			
Overnight soak				
FTP	Yes	Yes		
CFDS	Yes	Yes		
HFET x 3	Yes	Yes		four
30 min @ idle	Yes			
Prepare Vehicle for Owner				
Return Vehicle to Owner				

The organization of these vehicle testing modes into a single complete vehicle-test is shown in Table 1. Each of the three test-phases begins with an afternoon portion of testing in which only particulate emissions (no gaseous or fuel consumption) and particulate bulk samples are obtained. The afternoon portion begins with 30 minutes of 50 mph cruise with no sampling to stabilize the vehicle after fuel/oil change. As indicated in Table 1, the remainder of the afternoon follows a sequence of (1) 50 mph cruise, (2) CFDS, (3) 3x HFET; at this time the vehicle is placed in an overnight "cold soak" in preparation of the next mornings' FTP. The morning of testing begins with an 1979 FTP with 1981 diesel particulate sampling, followed by (1) CFDS, (2) 3x HFET, and (3) idle. Gaseous measurements are made for all the morning cycles with exception for the final idle period.

This test protocol generates 21 bulk particulate samples (30 cm x 50 cm) and 33 47 mm particulate emissions filters (not including back-up filters) from each complete vehicle test.

It was not possible to use a single reference oil for Phase III of each privately owned vehicle test considering the manufacturer's recommendations for the various vehicle types and the seasonal changes in Albany, New York. The third phase was thus run with oil and filter of vehicle manufacturer specification, selecting from either Castrol 30 W, 10W30, or 20W50.

Vehicle Sample Group - The 19 vehicles presently in the sample group are listed in Table 2, "Diesel Vehicle Test Roster," along with vehicle and test information including dynamometer road-load, date, odometer, and vehicle test identification number. Table 3 gives a summary of the 19 vehicles and 34 tests by manufacturer. This categorization does not separate vehicles by model year, drive train or engine calibration differences that may effect individual vehicle emissions. Fig. 2 shows the distribution of vehicle tests by odometer reading at each test point to indicate the range of vehicle mileage for tests on sample group vehicles. Only two vehicles have accumulated sufficient mileage and been tested enough times to provide a measure of mileage accumulation effects. Several different dynamometer road-load values were used to certify different vehicles produced by a given manufacturer employing a common engine. These variations in road-load may have impact on the emissions parameters grouped by "vehicle manufacturer" in subsequent tabulations.

Privately owned vehicles were solicited by letter to registrants of diesel vehicles in the Albany, N.Y. area. In consideration for the use of their vehicle for testing, vehicle owners receive the use of a loan car during the vehicle test, their vehicle is returned with two car wash tickets, a full fuel tank and an oil and oil filter change. Two project vehicles (cars #1 and 5) serve as the loan vehicles, and also are used for special test purposes, such as the large particulate sample collection work for chemical characterization mentioned previously. Two Oldsmobiles (cars #2 and 3) belong to the NYS Thruway Authority and

Table 2-Diesel Vehicle Test Roster

Project I. D. #	Make Year	Model	Engine Displace. (liters)	Transmission	Dynamometer Road Load	Dynamometer Inertia Weight	(Test No. TEST #) / (Month/Day/Year) / (Odometer) 1	2	3	4	5
1	VW 79	Rabbit	1.5 L	MA	7.3	2750	2 10/23/79 3,575	34 6/10/80			
2	Olds 79	Cutlass Cruiser	V8 5.7 L	A3	12.5	4000	1 9/27/79 3,991	7 1/22/80 11,679	18 4/21/80	30 7/30/80	
3	Olds 79	Cutlass Cruiser	V8 5.7 L	A3	12.5	4000	2 10/5/79 4,150	6 1/14/80	20	31	
4	Olds 79	98 Regency	V8 5.7 L	A3	12.5	4500	3 1/7/79 4,720	21 5/20/80			
5	Olds 79	Cutlass Cruiser	V8 5.7 L	A3	12.5	4000	4 10/30/79 5,017	25 6/2/80			
6	VW 80	Rabbit	1.5 L	MA	8.0	2250	8 1/30/80 4,225	33 8/18/80			
7	Cadillac 79	Eldorado	V8 5.7 L	A3	10.6	4500	9 2/6/80 11,111	29 7/22/80			
8	VW 78	Rabbit	1.5 L	MA	8.0	2250	10 2/25/80	30 6/23/80			
9	VW 79	Rabbit	1.5 L	MA	7.3	2250	11 3/3/80	37 6/30/80			
10	Dodge 79	D-15 Ritow-Diesel	5.8 L	A3	14.4	5500(+)	12 3/10/80				
11	VW 77	Rabbit	1.5 L	MA	8.0	2250	13 3/17/80	39 7/16/80			
12	M-B 77	240-D	2.4 L	MB	11.3	3500	14 3/26/80				
13	M-B 78	300 SD	3.0 L	A4	12.0	4000	15 3/31/80				
14	M-B 79	240 D	2.4 L	MB	12.0	3500	16 4/7/80				
15	Audi 79	5000	2.0 L	M3	11.0	3000	17 4/14/80	36 9/8/80			
16	Olds 79	Delta 88	V8 5.7 L	A3	13.3	4500	19 4/29/80				
17	Peugeot 79	504	2.1 L	MB	10.7	3500	22 5/27/80				
18	Olds 80	Cutlass Cruiser	V8 5.7 L	A3	12.5	4000	35 6/17/80				
19	M-B 1979	300-SD	I-5 3.0 L	A4	13.0	4000	32 8/11/80				

TABLE 3

Sample Group Vehicles by Manufacturer and Tests

Manufacturer	No. of Vehicles	Vehicle Tests
General Motors	8	18
Volkswagen	4	10
Mercedes-Benz	4	4
Audi	1	2
Peugeot	1	1
Mitsubishi	1	1
TOTALS	19	34

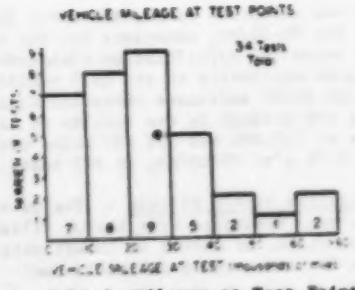

Fig. 2 - Vehicle Mileage at Test Points.

accumulate approximately 30,000 miles per year in normal administrative service, and provide the first project data on mileage accumulation effects.

Fuel and Oil - Vehicle owners are now encouraged to call to arrange for a test on their vehicle when it is due for an oil change, although for a few early tests the "as received" oil was not necessarily due for change. Vehicles delivered with 1/4 to 1/2 tank-full fuel are acceptable for testing but if delivered with less than this amount they may run out of fuel during the first test phase, and thus are filled with commercial (non AEL reference) fuel prior to the beginning of the vehicle test.

When ready for testing, three fuel samples are taken from the tank. One sample is analyzed for: API gravity, specific gravity, heating value, carbon, hydrogen, nitrogen, sulfur, oxygen, ash, complete distillation, and cetane index by Fuel Engineering, Thornwood, N.Y. A second sample is analyzed for asturates, olefins, and aromatics using ASTM D-1319 by the Source, Fuels, and Molecular Chemistry branch of EPA/RTP. A third portion is kept at AEL for project reference. A sample of "as received" crankcase oil from each vehicle is also collected and kept at AEL for project reference. Fuel analyses data for the reference fuel stored at AEL for vehicle testing are given in Appendix A.

SAMPLE HANDLING PROCEDURES

Weighing of Filters - The 47 mm diameter Pallflex filters were placed in a constant humidity chamber for at least 24 hours before each weighing. The temperature was maintained at 18 to 21°C with a relative humidity range of 35 to 60%. The equilibrated filters were weighed on a Mettler ME 30 microbalance in micrograms. 50 cm x 50 cm filters were not equilibrated to any standards but were kept in areas where the temperature was usually about 20°C and the relative humidity in the 30 to 50% range. Tare weights were taken by carefully rolling the filters into approximately 15 cm cylinders and placing them on a semicircular weighing pan in a Mettler H10T balance. The balance doors were kept open to accommodate the length of the rolled filters. The balance itself was in a large five-sided box to reduce air currents and the weighings were performed in a balance room without air circulation. Gross weights were taken after the filters had been folded into quarters and were performed in a normal manner. Weights were taken to the nearest milligram with the uncertainty in the net weight estimated to be about ± 5 mg.

Extraction of Filters - The 50 cm x 50 cm filters used for bulk particle collection were extracted with dichloromethane to obtain the SOF as a percentage of the particulate mass on the 50 cm x 50 cm filters. The filters were folded and placed in 50 mm Soxhlet extractors without thimbles. Extraction was carried out with approximately 300 ml of dichloromethane (Burdick and Jackson Laboratories, Distilled in Glass™ grade) for 24 hr at three-four cycles per hour.

The extracts were filtered into 500 ml flasks by vacuum through an 0.2 µm Fluoropore filter (Millipore FGLP) to remove any particles which may have carried over. The solution was heated to boiling before filtering and the filtering apparatus was kept hot during filtration to prevent precipitation of any sparingly soluble material on the filter. The 500 ml flasks were rotary evaporated to about 75 ml volume and the sample transferred to acid and solvent washed, dried and tared 125 ml borosilicate glass bottles with Teflon lined caps. These bottles were then partially immersed in a warm water bath while attached to the rotary evaporator to remove the remaining solvent to yield the dried particulate extract. Final weighing provides by difference the mass of extract from which the SOF percentage was computed directly. Bottles with dried extract are then stored at -80°C until needed for bioassay.

Each vehicle test results in 21 50 cm x 50 cm filters, 15 of these have been selected for extraction, the remainder kept in -80°C storage. The six unextracted filters are the CFDS and HFET filters from the afternoon portion of each phase. Four vehicle tests for which gaseous and particulate emissions (g/mi) are available at this writing are yet to be extracted (tests 29, 32, 33, 34 - see roster Table 2). This leaves about 450 discrete 50 cm x 50 cm filters that have been extracted as the SOF data base in this paper.

GASEOUS AND PARTICULATE EMISSIONS

Gaseous Emissions and Fuel Economy - Average emissions (gaseous and particulate) and fuel

TABLE 4

Emissions and Fuel Economy Results - Average All Phases
(g/mi and mi/gal)

Car No.	Phase	Model	FTP					HFET				
			HC	CO	NO_x	Particulate	Mi/gal	HC	CO	NO_x	Particulate	Mi/gal
1	81	[illegible]	0.37	1.19	0.84	0.40	41.9	0.25	0.99	0.77	0.34	55.7
2	81	[illegible]	0.39	1.06	1.01	0.76	38.3	0.28	0.62	1.03	0.26	57.2
3	80	[illegible]	0.27	1.08	1.08	0.76	38.4	0.24	0.88	1.22	0.28	56.3
4	80	[illegible]	0.26	1.06	1.03	1.07	38.3	0.21	0.88	1.20	0.39	56.3
5	80	[illegible]	0.31	1.08	1.08	0.91	38.2	0.24	0.87	1.20	0.37	55.9
6	80	[illegible]	0.24	1.08	0.99	0.87	42.8	0.21	0.83	0.92	0.30	58.3
7	80	[illegible]	0.28	1.01	1.09	0.84	38.3	0.27	0.87	1.14	0.34	56.3
8	80	[illegible]	0.24	1.09	0.88	0.82	40.9	0.18	0.88	0.76	0.32	56.2
9	80	[illegible]	0.22	0.98	0.98	0.93	43.4	0.20	0.76	0.89	0.39	57.4
10	80	[illegible]	0.28	1.00	0.98	0.86	38.5	0.24	1.00	2.17	[illegible]	[illegible]
11	80	[illegible]	0.28	1.06	0.98	0.87	43.2	0.27	0.84	0.88	0.37	58.1
12	80	[illegible]	0.28	1.09	1.09	0.88	37.3	0.25	0.98	1.08	0.38	[illegible]
13	80	[illegible]	0.31	1.01	1.07	0.87	38.9	0.28	0.97	1.09	0.43	57.-
14	80	[illegible]	0.32	1.09	1.03	0.88	38.8	0.31	0.88	1.20	0.39	55.-
15	80	[illegible]	0.28	1.09	1.03	0.89	38.-	0.26	0.88	1.14	0.38	56.-
16	80	[illegible]	0.42	1.09	1.07	0.88	38.9	0.36	0.88	1.13	0.42	56.-
17	80	[illegible]	0.33	1.04	0.99	0.82	38.8	0.28	0.91	0.83	0.34	58.9
18	80	[illegible]	2.80	2.80	0.75	0.80	30.0	2.32	2.11	0.59	0.80	32.-
19	80	[illegible]	0.27	1.08	1.08	0.90	38.5	0.27	0.98	1.27	0.39	56.9

economy results by individual vehicles are given in Table 4 for the FTP and HFET cycles. The results are averaged over all three test-phases and with repeat tests (if any) to give a single value for each sample group vehicle. Although no detailed partition of FTP particulate emissions by individual bags is presented in this paper, results from 99 FTP runs have averaged 44% greater particulate emission in bag 1 (cold start at 72°F) than for bag 3 (hot start at 72°F).

Car #10 is a Dodge pickup equipped with a Mitsubishi diesel engine, which was certified by the manufacturer as a heavy duty engine. This limited-production vehicle is in New York State Thruway service and has been included in this study to increase the variety of engines in the project data base. One 1980 General Motors 5.7 liter diesel vehicle has been tested to date (car #18, tested at 4,000 miles) and the results are probably quite abnormal. This engine has been significantly changed for 1980, including new hardware (EGR, etc.) and new calibrations. The effect of these changes on in-use emission is unknown at this time. The vehicle ran very poorly throughout testing as characterized by excessive engine rocking at all but acceleration modes. The cause of this was not determined, but AEL technicians did recommend to the owner that the vehicle be returned to the dealer for check-out. The owner did have the dealer examine the vehicle and new injector lines were installed; however, at 12,000 miles the vehicle runs roughly and will be returned for further service.

With the exception of the two above mentioned vehicles, the HC/CO/NO_x emissions for the sample group are below the certification standards of 1.5/15/2. g/mi applicable to the 1979 vehicles. The inherent low HC/CO emissions characteristics of the diesel are evident in the results on car #11, with tests at 120,000 and 133,000 miles averaging 0.33/1.04/0.75 g/mi HC/CO/NO_x in FTP tests.

Particulate Backup Filters - Twelve vehicle tests have been conducted with backup filters for every 47 mm filter collected to investigate their importance for the various vehicles, fuels, and

TABLE 5 Particulate Emissions Reproducibility

Average Particulate Emissions (g/m) Differences Between Duplicate Cycles

[% difference ± 1 σ]

	Obs. in each Average	Phase I		Phase II		Phase III		Three Phase Average	
		CFDS	HFET	CFDS	HFET	CFDS	HFET	CFDS	HFET
All Tests	34	1.0%± 8.8	1.2%±10.6	0.1%±10.2	1.8%±8.1	1.2%± 9.2	0.8%± 8.0	0.7%	1.3%
General Motors	16	0.5%± 7.2	2.7%± 9.0	-1.2%± 7.5	0.0%±6.3	0.6%± 5.8	1.3%± 5.5	0.8%	1.7%
Volkswagon	10	1.0%± 12.3	1.8%±14.9	3.5%±15.7	0.6%±7.2	2.9%±13.4	3.7%± 9.2	2.5%	2.0%
Mercedes-Benz	4	1.8%± 8.3	-5.7%± 7.9	-0.3%± 5.6	-3.6%±3.4	3.1%± 8.8	0.2%± 2.0	1.7%	-3.2%

Note: % > 0 indicates first cycle > second cycle
% < 0 indicates first cycle < second cycle

driving schedules. Average backup filter contribution to the combined mass (primary plus backup) was 1.65% (σ = 0.95) for 295 individual test segments (i.e. 295 separate filters). The backup contribution for FTP testing was 2.66% (σ = 1.6) for 96 observations. The group of filters with the highest backup contribution came from the idle test mode, where 35 tests gave backup contributions that averaged 5.68% (σ = 5.75). Prior to the time of this finding for the idle test, the CVS flow setting had been left at a constant value for all cycles for a given vehicle. This resulted in the idle sampling being conducted at a significantly higher exhaust dilution ratio than the other cycles used. Backup filters from idles under these conditions frequently gave higher absolute mass loadings than for those from other (higher speed) driving cycles. The collection efficiency for filter media is known to be increased by previously deposited particulate on the filter, and in the dilute idle exhaust the primary filter never received sufficient mass loading for this efficiency enhancement to occur. A subsequent decision to decrease the dilution ratio for idles increased the primary filter loadings and decreased the backup filter loading.

In contrast to the general finding that backup filters collected a small percentage of the total particulate, tests on a Peugeot (car #17) exhibited distinctly different results. Particulates from this vehicle did not mat on the face of the filter media but penetrated to give a grey appearance on both the front and back of the filter. Backup filters on 30 Peugeot tests averaged 9.4% of the total particulate mass. In 7 Peugeot tests, two 50 cm x 50 cm sheets were used in series in the same holder, and the second 50 cm x 50 cm filter contained an average of 9.5% of the total. This observation parallels that of the USEPA for a Peugeot vehicle (36, 37). Backup filters have been discontinued for all vehicles except the Peugeot, but variations in sampling conditions and vehicle type can impact the need for backup filters.

Particulate Emissions Reproducibility - Within each test phase the duplicate CFDS and HFET cycles performed in the afternoon and again the following morning can be compared to determine the stability of particulate emissions during the given phase. Table 5 shows the reproducibility for these two driving cycles for each vehicle test phase for all 34 tests and also by vehicle make subcategory. On the average, the reproducibility is in the 1-3% range, but the standard deviations for the sample are in the 5-15% range. The Volkswagons show a larger standard deviation than other groups, possibly due to their manual transmissions in contrast to the automatic transmissions on most other sample group vehicles.

Filter Pair Correspondence - The two 47 mm diameter filter collection systems used for the gravimetric measurements by the 1981 FTP particulate standards are routinely operated simultaneously during all CFDS cycles as an on-going check of this system's correspondence and data integrity. For all 111 CFDS tests, the mean absolute difference of these simultaneous measurements was 0.015 ± 0.013 g/mi which is 3.3% ± 2.8% of the overall average CFDS particulate emission rate of 0.457 g/mi, indicating acceptable performance of these systems.

The 50 cm x 50 cm bulk particulate collection system was designed to obtain significant quantities of particulate for chemical and biological analysis and as such is different in design and was not optimized to insure strict gravimetric correspondence with the 47 mm diameter filter systems discussed above. The mean absolute difference of the 50 cm x 50 cm particulate emission rate referenced to the 47 mm system was -0.04 ± 0.02 g/mi, indicating a small but systematic lower collection rate by the bulk particulate system.

In consideration of the standard deviations, the small percentage differences indicates no systematic change in emissions between the duplicate cycles, while individual tests can be expected to exhibit 5 - 15% positive and negative deviations for duplicate tests.

Particulate Emissions Results - Average particulate emissions (g/m) are presented in Fig. 3 for FTP, HFET, CFDS, and 50 mph cruise driving cycles for General Motors, Volkswagon, and Mercedes-Benz vehicles groupings. Values in Fig. 3 include results from all three test phases. The Volkswagon results are relatively constant for all

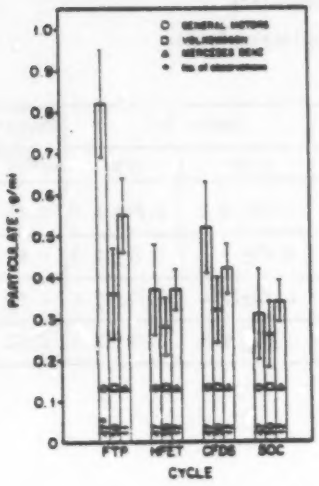

Fig. 3 - Particulate Emissions, g/mi, by Test Cycle and Vehicle Group.

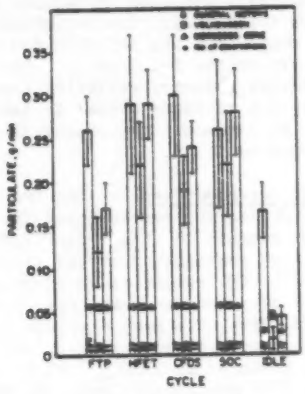

Fig. 4 - Particulate Emissions, g/min, by Test Cycle and Vehicle Group.

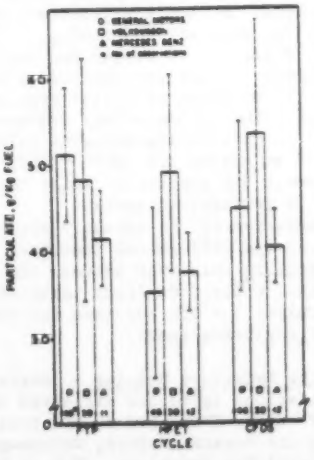

Fig. 5 - Particulate Emissions, g/Kg Fuel, by Test Cycle and Vehicle Group.

four driving cycles, whereas the General Motors and Mercedes-Benz groups show higher emissions during the FTP than for the other cycles. For each of the three vehicle groups the CFDS particulate emission levels are higher than the value for the respective groups in HFET and 50 mph cruise. This may be due to the more numerous acceleration/deceleration modes of the CFDS compared to the HFET and 50 cruise cycles.

Particulate emissions for each driving cycle have been computed on an emission per unit time basis to facilitate comparison of idle emissions with those from other driving cycles. Fig. 4 shows the results of the three vehicle groupings in grams particulate/minute for the various vehicle operating cycles (all test phases included). Idle emission rates for Volkswagon and Mercedes-Benz vehicles are significantly lower than for General Motor vehicles; the time emission rate of particulate for the General Motors vehicles is only a factor of 1.7 lower for the idle mode than the average of other modes tested, whereas the Volkswagon and Mercedes-Benz vehicles have comparable ratios of 9.4 and 5.4 respectively.

The Volkswagon and Mercedes-Benz follow almost identical patterns with Mercedes-Benz consistently higher by about 0.06 g/min except for the idle where the difference is much smaller. The ratios of Mercedes-Benz to Volkswagon are 1.46, 1.30, 1.31, 1.25 and 2.46 for the FTP, HFET, CFDS, 50 C and IDLE, respectively. The Volkswagons and Mercedes-Benz also differ from the General Motors vehicles in that there is an increase in particulate emissions per minute as the average speed of the driving cycle increases, not observed with the GM vehicles.

Particulate emissions have been calculated on a fuel specific basis for those driving cycles in which gaseous emissions are measured (FTP, HFET, and CFDS). Fig. 5 gives these results as particulate grams per kilogram fuel consumed for the three vehicle groupings used previously (all test phases included). Fuel specific particulate ranges average from 3.5 to 5.5 g/Kg fuel for the FTP, HFET and CFDS cycles with the three vehicle groupings. Volkswagon vehicles are relatively constant for the three cycles (4.8 to 5.4 g/Kg fuel), as are the results on Mercedes-Benz vehicles (3.7 to 4.2 g/Kg fuel). General Motors vehicles exhibit larger differences among cycles (3.5 to 5.1 g/Kg fuel) with the lower value of the range found on HFET driving.

Soluble Organic Fraction (SOF) Results - The term soluble organic fraction (SOF) is applied to that portion of the particulate which is extracted by dichloromethane. Fig. 6, 7, 8, show the SOF data in terms of %, g/mi and g/Kg fuel respectively. In Fig. 6 it can be seen that the Mercedes-Benz shows very little variation in SOF percentage among driving cycles. The Volkswagon rate is almost constant for the driven cycles but increases very sharply for the idle. The General Motors vehicles show a definite cycle effect and have higher values for all cycles except the idle.

Fig. 7 shows this data in g SOF/mi. In this comparison no vehicle group shows any consistent value but there is a common trend in the relative values for each cycle. In each case the g SOF/mi decrease in the order FTP > CFDS > HFET

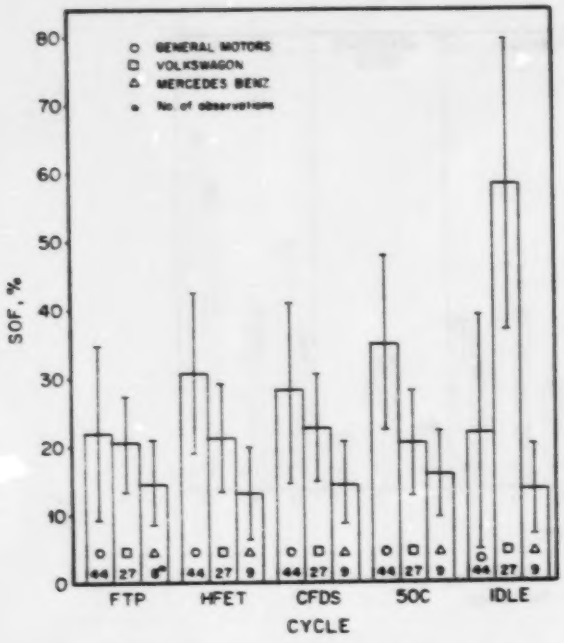

Fig. 6 - Soluble Organic Fraction, %, by Test Cycle and Vehicle Group.

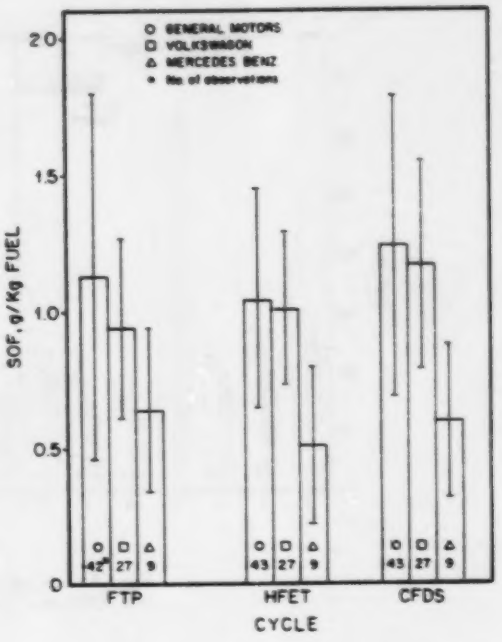

Fig. 8 - Soluble Organic Fraction, g/Kg Fuel, by Test Cycle and Vehicle Group.

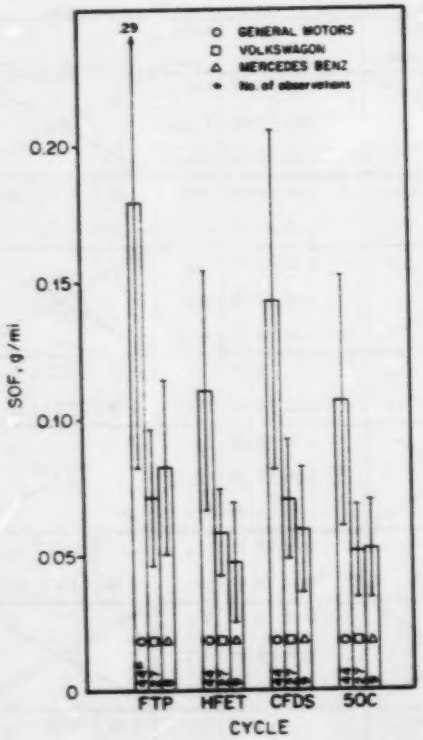

Fig. 7 - Soluble Organic Fraction, g/mi, by Test Cycle and Vehicle Group.

> 50 C. The General Motors vehicles are again the highest and are even more greatly separated from the other vehicles than in Fig. 6. This is caused by the higher g/mi particulate emission rate for these vehicles. The idle value for Volkswagon is now comparable to that for Mercedes-Benz due to the very low g/mi particulate emission rate.

Fig. 8 shows the SOF in fuel specific terms, i.e. g SOF/Kg fuel for the three cycles for which gaseous emission data were collected. The trend here is similar to that for SOF in g/mi and %. In each case the General Motors vehicles have the highest values followed by Volkswagon and Mercedes-Benz. In this case, however, the General Motors vehicles have values close to those for the Volkswagon; Mercedes-Benz results are considerably lower. No consistent cycle-to-cycle variation is evident.

SOF (%) average results are given in Fig. 9 for all tests on each individual car in the sample group. Both FTP and HFET results are shown with the standard deviation indicated. Vehicles with several tests at various mileage test points show larger deviations since the SOF (%) has changed from test to tests. The unique SOF % results for the single 1980 Oldsmobile (car #18) are evident. The Peugeot tests (car #17) also show high SOF (%) values.

Particulate and SOF Emissions Variability - The three fuel/oil test conditions can be compared in terms of particulate emissions and particulate extract (SOF) emissions to see if any systematic

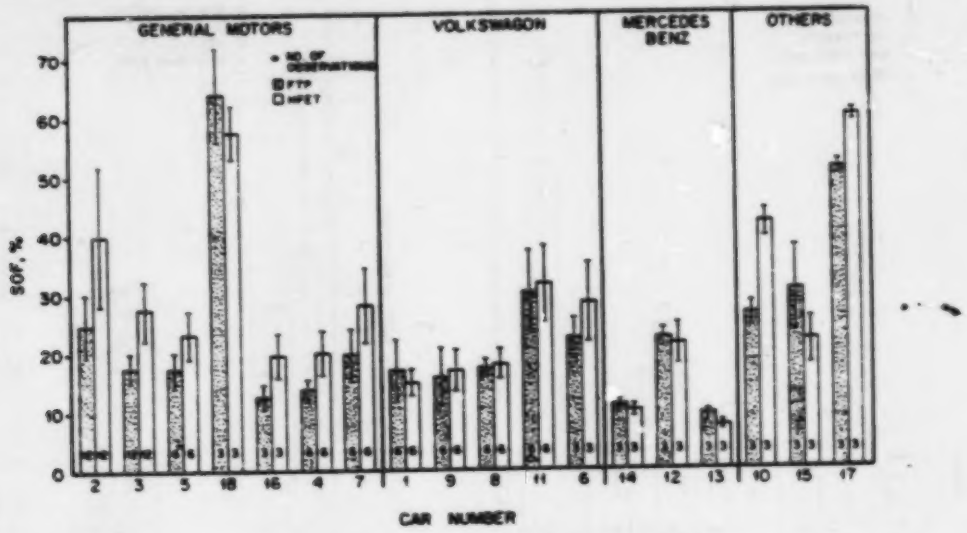

Fig. 9 - Soluble Organic Fraction, %, for FTP and HFET for Individual Vehicles.

Table 6-Particulate and SOF Emissions: Reproducibility Within Test Phases and Variability Among Test Phases

$$R = \left[\frac{(\text{gram/mile})_A}{(\text{gram/mile})_B}\right]$$

A : First Test
B : Second Test

			CFDS						HFET					
			Particulate (g/m)			SOF (g/m)			Particulate (g/m)			SOF (g/m)		
			$\bar{R}$	σ	obs<1 / obs>1	$\bar{R}$	σ	obs<1 / obs>1	$\bar{R}$	σ	obs<1 / obs>1	$\bar{R}$	σ	obs<1 / obs>1
ALL	Reproducibility in each vehicle test phase	I	1.01	0.09	14/20				1.01	0.11	19/15			
		II	1.00	0.10	15/18				0.98	0.08	17/16			
		III	1.01	0.09	18/16				1.01	0.08	13/21			
	Phase-to-Phase Variability	I/III	0.99	0.11	19/15	1.02	0.21	13/17	1.04	0.10	14/20	1.06	0.24	18/12
		II/III	1.01	0.13	15/18	1.03	0.20	17/12	1.02	0.10	12/21	1.01	0.21	16/13
GENERAL MOTORS	Reproducibility in each vehicle test phase	I	1.00	0.07	8/8				1.03	0.09	7/9			
		II	0.99	0.08	6/9				1.00	0.06	6/9			
		III	1.01	0.06	8/8				1.01	0.05	6/10			
	Phase-to-Phase Variability	I/III	1.00	0.08	9/7	0.97	0.24	10/5	1.02	0.08	9/7	0.96	0.23	11/4
		II/III	1.00	0.07	7/8	0.95	0.19	10/4	1.01	0.09	6/9	0.96	0.20	9/5
VOLKSWAGON	Reproducibility in each vehicle test phase	I	1.01	0.12	4/6				1.02	0.15	6/4			
		II	1.04	0.16	4/6				1.01	0.07	4/6			
		III	1.03	0.13	6/4				1.04	0.09	2/8			
	Phase-to-Phase Variability	I/II	0.99	0.13	7/3	1.05	0.17	2/7	1.08	0.13	2/8	1.16	0.22	4/5
		II/III	1.00	0.16	5/5	0.98	0.20	6/2	0.99	0.09	5/5	0.98	0.20	6/3
MERCEDES-BENZ	Reproducibility in each vehicle test phase	I	1.02	0.08	1/3				0.96	0.03	3/1			
		II	1.00	0.06	2/2				0.94	0.08	3/1			
		III	0.97	0.09	2/2				1.00	0.02	2/2			
	Phase-to-Phase Variability	I/III	0.96	0.17	1/3	1.14	0.07	0/3	1.10	0.10	2/2	1.13	0.14	1/2
		II/III	0.98	0.03	3/1	1.16	0.17	1/2	1.00	0.09	3/1	1.15	0.24	0/3

variations occur in the three test-phases. First, however, a measure of the repeatability for the emissions within a given test condition must be established. Since two CFDS and HFET tests are performed for each of the three fuel/oil combinations, comparisons of particulate emissions (g/mi) for these repeated cycles are easily obtained. For each pair of repeated cycles, the ratio of particulate (g/mi) is computed. Table 6 shows average values, standard deviations and number of observations greater than and less than unity for CFDS and HFET cycle ratios. These results are presented for all tests as one group, and also by vehicle type. Results are given in each case for all three vehicle test phases. For all 34 vehicle tests combined the ratio for repeated CFDS and HFET tests is very nearly unity, but the standard deviation is about 10% (.10 in Table 6). This variation in repeated measurements at one fuel/oil condition impacts the magnitude of differences among phases that can be resolved.

Examination of the phase-to-phase variability for all tests in one vehicle group shown in Table 6 shows these ratios and standard deviations to be comparable to the repeated measurements within a single phase, suggesting little influence on total particulate emissions of the fuel/oil combinations. This is not totally unexpected in that the reference fuel is merely a lot of commercially available fuel.

Repeatability checks within a test-phase for SOF (g/mi) determinations are not available since only one of the two repeated filters has been extracted at this time. Phase-to-phase comparisons of SOF (g/mi) emissions are shown, and for both CFDS and HFET tests show slightly higher SOF emissions in phases I and II than for phase III (~ 4%). The standard deviation for SOF is considerably larger than for the total particulate (~ 20%) so the systematic change among test phases is unlikely to be a definite trend.

Vehicle sub-groups in Table 6 can be examined to seek more detailed phase-to-phase and repeatability information. The number of observations less than and greater than unity provide a rough indication of the shape of the distribution from which each entry was obtained, and, of course, the number of observations available from which the results were computed.

Phase-to-phase variabilities for the sample group as a whole are given in Table 7 for a variety of specific driving schedules. Particulate and SOF data are both shown as ratios using the results from phase III as a common denominator in each given vehicle test. Little systematic difference is evident, but the standard deviation in FTP tests is seen to be larger than for other cycles (20% vs. 11%) with the exception of the idle mode. Phase-to-phase SOF variations result in roughly the same standard deviations for all cycles.

For the range of test conditions encountered up to this point the data do not indicate any significant systematic variation in either particulate or SOF emissions, although the magnitude of standard deviations clearly show that a single test on a particular vehicle may result in significant variations in even a repeated test cycle, as well as for a new fuel/oil combination.

Mileage Accumulation Effects - As an aid in assessing the effects of mileage accumulation on vehicle emissions two 1979 Oldsmobile Cutlass Cruisers were purchased for use by the New York State Thruway Authority. These two vehicles are operated under essentially the same highway conditions in administrative service; and each accumulates about 2,500 miles/month (30,000 miles/year). These vehicles are being tested at 8,000 to 10,000 miles and have now been each tested four times. Results for particulate emissions and SOF are shown in Fig. 10. The values shown in Fig. 10 are averages of all applicable test cycles and phases at each mileage test point. Car #5, another Cutlass Cruiser stationed at AEL as a control test vehicle and loan car for access to privately owned vehicles, has also

Table 7-Particulate and SOF Emissions: Variability Among Test Phases by Driving Cycle.

All vehicle tests taken as one group of measurements, Emission ratios computed:

$R = \left[\frac{\text{(g/m) phase I}}{\text{(g/m) phase III}}\right]$ and $R = \left[\frac{\text{(g/m) phase II}}{\text{(g/m) phase III}}\right]$

Ratios for all tests compared to obtained mean ($\bar{R}$) and standard deviation (σ)

Vehicle Test Cycle =		FTP		HFET-1st		HFET-2nd		CFFS-1st		CFFS-2nd		50 mph		Idle	
Ratio of Phases =		I/III	II/III	I/III	II/III	I/III	II/III	I/III	II/III	I/III	II/III	I/III	II/III	I/III	II/III
Total Particulate (g/m)	$\bar{R}$	1.001	0.991	1.046	0.998	1.043	1.024	0.994	0.989	0.995	1.006	1.037	1.047	1.047	1.091
	σ	0.24	0.17	0.12	0.07	0.10	0.10	0.12	0.09	0.11	0.13	0.12	0.12	0.34	0.21
	obs<1/obs>1	12/15	20/12	16/18	16/17	14/20	12/21	21/13	16/17	19/15	15/18	15/19	12/21	11/21	6/26
Particulate SOF (g/m)	$\bar{R}$	1.025	0.961	-	-	1.057	1.013	-	-	1.024	1.029	1.058	1.069	1.04	1.074
	σ	0.35	0.23	-	-	0.24	0.21	-	-	0.21	0.20	0.35	0.29	0.54	0.29
	obs<1/obs>1	19/11	17/11	-	-	18/12	16/13	-	-	13/17	17/12	16/14	11/18	12/17	12/15

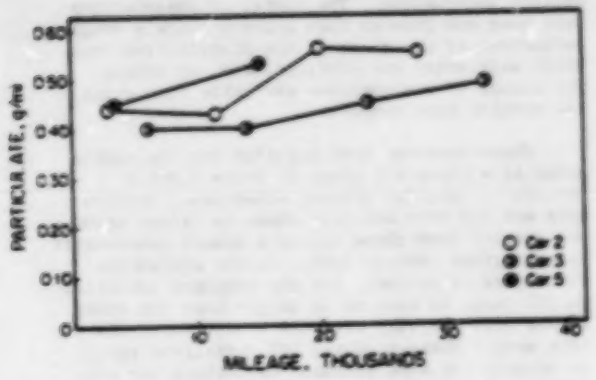

Fig. 10 - Mileage Accumulation Effects on Particulate Emissions, g/mi, for Oldsmobile Cutlass Cruisers.

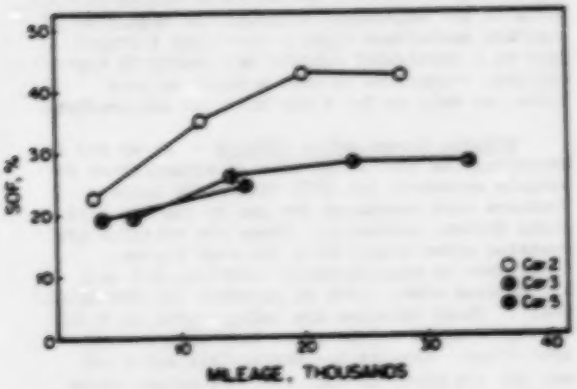

Fig. 11 - Mileage Accumulation Effects on Soluble Organic Fraction, %, for Oldsmobile Cutlass Cruisers.

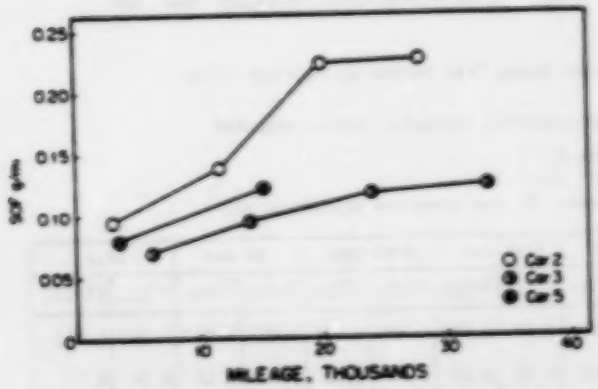

Fig. 12 - Mileage Accumulation Effects on Soluble Organic Fraction, g/mi, for Oldsmobile Cutlass Cruisers.

been included in these figures for comparison. Fig. 10 shows a general, although nonsystematic, increase in particulate emissions with increasing mileage which averages about 0.05 g/mi increase per 10,000 miles. It is uncertain whether this trend will continue in further tests.

Fig. 11 shows that the % SOF also increases with accumulated mileage but is more systematic than the particulate data. The % SOF generally increases rapidly for the first approximately 20,000 miles and then levels off for an as yet undetermined period. Vehicle-to-vehicle differences are very apparent in this Figure where Car #2 shows consistently higher and more rapidly increasing % SOF than the other two vehicles. Fig. 12 shows g/mi SOF and demonstrates a similar trend for all three vehicles with Car #2 again higher than the other two vehicles. The combination of increased particulate emissions and increased % SOF leads to an average increase in SOF, g/mi, of 80% for each 10,000 miles accumu-

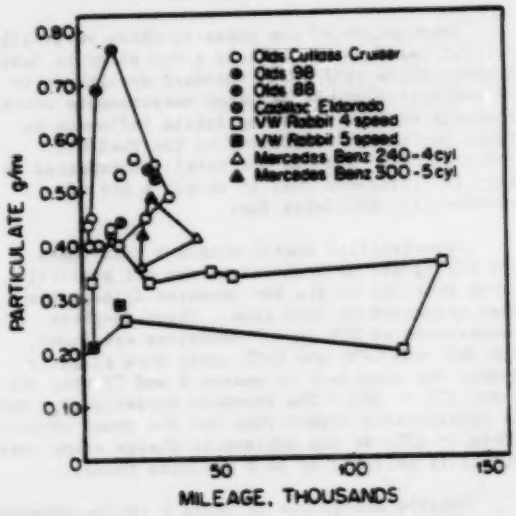

Fig. 13 - Mileage Accumulation Effects on Particulate Emissions, g/mi, for General Motors, Volkswagon and Mercedes-Benz Vehicles.

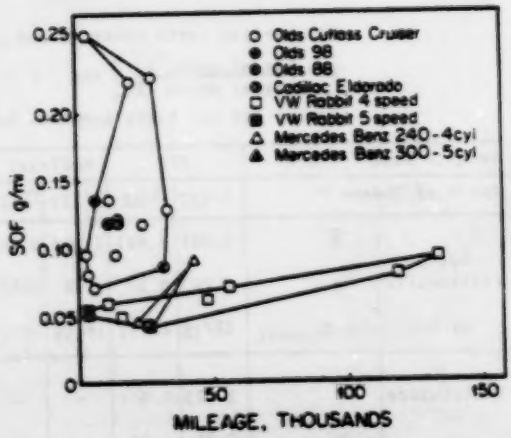

Fig. 14 - Mileage Accumulation Effects on Soluble Organic Fraction, g/mi, for General Motors, Volkswagon and Mercedes-Benz Vehicles.

lated by Car #2 up to 20,000 miles. In general the three vehicles are much more similar to each other at low mileage than at higher mileage.

In Figs. 13 and 14 each major vehicle class i.e. General Motors, Volkswagon and Mercedes-Benz is represented by a different type of symbol and a solid line delineates the envelope which contains all the points for a given vehicle class. The vehicle classes show some overlap but are generally well resolved with General Motors showing the highest particulate emissions followed by Mercedes-Benz and then Volkswagon. More detailed comparisons are difficult as there is a lack of high mileage data except for two tests on one Volkswagon at this time. All the Cutlass Cruisers and the General Motors 5.7 liter V8 diesel vehicles in general fall into a relatively discrete envelope of values as compared to Volkswagon and Mercedes-Benz vehicles.

Figs. 15, 16, and 17 show particulate and SOF data for Volkswagon Rabbits only. Fig. 15 shows particulate emissions for five vehicles each of which was tested twice. In all but one case the particulate emissions increased markedly (0.07 g/mi/10 K mi) between tests although further tests are needed to determine if this is a mileage related change or random variation due to other unknown factors. Fig. 16 which shows % SOF indicates that particulate emissions and % SOF change in opposite directions. This trend would tend to keep g/mi SOF fairly constant but as is apparent in Fig. 17 the increase in particulate emissions more than compensates for the decrease in % SOF and there is an increase in absolute amount of material extracted. The apparent decrease in SOF g/mi for car #5 may have been caused by the correction of a significant injector timing error performed between tests.

No Mercedes-Benz vehicles have been tested more than once and the four tests had similar mileages. Therefore, no mileage accumulation data will be presented.

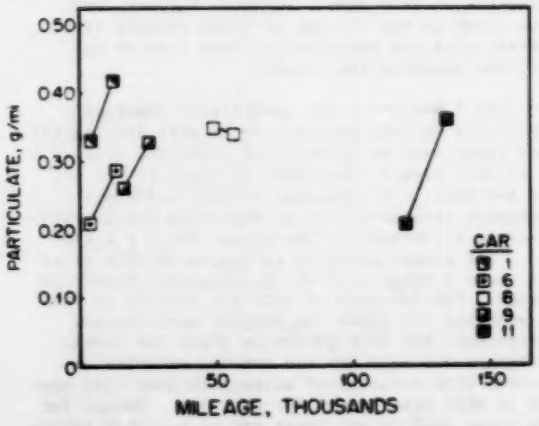

Fig. 15 - Mileage Accumulation Effects on Particulate Emissions, g/mi, for Volkswagon Rabbits.

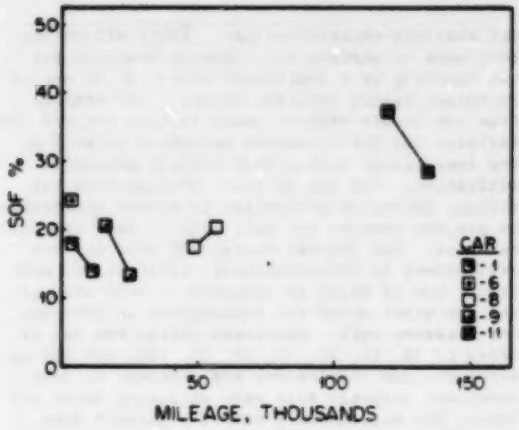

Fig. 16 - Mileage Accumulation Effects on Soluble Organic Fraction, %, for Volkswagon Rabbits.

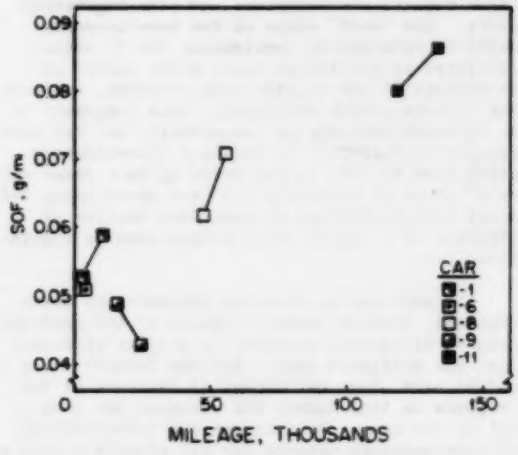

Fig. 17 - Mileage Accumulation Effects on Soluble Organic Fraction, g/mi, for Volkswagon Rabbits.

AMES BIOASSAY OF SOF SAMPLES

Ames Test Procedures - The bacterial mutagenesis plate incorporation assay according to the method of Ames (38) has given positive indications of mutagenic chemical species in SOF but since the chemical identity of these compounds are not fully known, the assay itself is presently the only available tool for routine detection of their presence. The potential for mutagen artifact from sampling in the dilution tunnel cannot be presently ruled out, as evidence of nitro-PAH mutagens in SOF is advanced (28, 39, 40). Until the chemical understanding is more fully developed, interactions of vehicle emission products and sampling and handling artifacts cannot be separated, and therefore the sampling procedures are not able to quantify mass emissions of the critical species. Thus, the mutagenic potency of SOF samples are given here to provide a relative measure of the project variables for internal comparisons, and are

not absolute emission values. Every effort has been made to perform all steps of sampling and SOF handling in a consistent manner so as not to introduce random artifact errors. SOF samples from the in-use vehicle tests in this project are analyzed for dose/response mutagenic potency by the Ames tester strain TA98 without metabolic activation. The use of other strains with and without metabolic activation have been undertaken on project samples but only TA98(-) data are included here. The freezer stored SOF extracts are redissolved in dichloromethane, split in two portions, one of which is returned to cold storage and the other dried and redissolved in DMSO for the bioassay work. Duplicate plates are run at doses of 10, 20, 30, 40, 50, 75, 100, and 200 ug. Straight-line regressions are obtained to dose/revertant response data sets utilizing first all doses, and subsequently with the highest dose eliminated from the regression, and successively with the two highest doses eliminated, etc. to look at the dose/response data for toxic effects that cause a plateau or fall-off of revertant counts with increasing dose. Appendix B contains three typical dose/response and data regression plots. The "best" slope of the dose/response curve is selected by considering the R^2 value, intercept of the fitted line, total number of revertants at the highest dose selected, and number of data points available. This judgement is by no means absolute or unequivocal, but has been applied consistently to obtain a (revertants/u grams dose of SOF) in the 0-200 ug dose range with an R^2 value of typically 0.90 and above using 5-8 doses with duplicates at each dose entered as separate data (total 10-16 points used in regression).

In addition to chemical standards to check response, freezer stored aliquots of SOF obtained from an Oldsmobile operated in project reference fuel are routinely used. For the TA98(-) tests reported here, the coefficient of variance of the response to this diesel SOF standard has been ± 23% of the mean. To increase the comparability of dose/response results for all samples within a given vehicle test, the 15 SOF samples from each vehicle test are run as a group on a single day. Two sets of SOF samples were analyzed in April of 1980, and reanalyzed in September. Table 8 shows the ratio of slopes obtained for each SOF sample for which duplicate runs were available, and provides a guide for interpreting the relative changes. The specific activity for all samples was about 25% reduced in the September analyses. Degradation of mutagens cannot be completely ruled out as contributing to these observed decreases, but bacterial response variations are considered more likely in view of the variable response to standards. The mutagenic potency of SOF samples are best considered as relative indicators and not absolute values, and large variations in specific activity (revertants/ug) are easily accumulated between the preparation of the SOF samples and the choosing of a slope from a SOF dose - revertant colony response curve.

Table 8-Change in Ames Response with Six Months Between Assays.

SOF Samples Assay on TA98(-)

Table Entries are Ratio of (revertants/mi) April / (revertants/mi) Sept.

	Test Phase	55 mph Cruise	FTP	CVS4	MVMA
Vehicle/Fleet	1	1.4	0.8	1.0	1.
Vehicle #3	2	1.6	0.8	1.5	1.5
Project Test #2	3	1.0	0.9	1.6	1.4
Average		1.3	0.8	1.4	1.3
Vehicle/Fleet	1	1.2	0.9	2.0	1.3
Vehicle #3	2	1.3	1.1	1.8	1.3
Project Test #2	3	1.0	1.7	1.3	1.8
Average		1.2	1.2	1.7	1.5

Average = 1.3, or Activity in September assay is 77% of April

Ames Test Results - The quantitative results of the Ames test are usually expressed simply as revertants/ug sample. In this case, where the composition of the sample (i.e. the SOF) is unknown, the amount of bio-active material in the sample is unknown. Therefore, the discussions below will generally consider mutagenic activity in terms of experimentally measurable parameters, i.e. grams of particulate, miles and Kg fuel consumed (Some data in terms of revertants/ug SOF are found in Fig. 24). The twelve sets of data below represent results of four tests each on two Oldsmobile Cutlass cruisers and one test each on an Oldsmobile 98, a Cadillac Eldorado, a Volkswagon Rabbit and a Mercedes Benz 300 CD; and are all the data available at this writing. The data for the results of the Ames test in terms of the measured slope of the dose-response curve and the derived parameters are presented in Appendix C. Except for the results on the Volkswagon Rabbit and Mercedes-Benz, all data are those of the September 1980 assay.

The amount of mutagenic activity may be expressed as revertants per gram particulate so as to remove, at least partially, the effects of changes in % SOF which may be caused by fuel and/or lubricant dilution of the extract. Revertants/g particulate (reported in units of 10^6 revertants/g which is equivalent to 1 revertant/ug) are equal to the slope of the dose response curve (rev/ug) times the % SOF divided by 100. These data are presented in Figures 18 and 19 for Car 2 and Car 3 respectively and for Cars 5, 8, 9 and 15 in Fig. 20. These twelve sets of data are the only results available at this time. In these figures, each data point is the average of three vehicle test phases, with the exception of Test 1 which had only two complete test-phases.

Car 2 and Car 3 are essentially identical Oldsmobile Cutlass cruisers which were each tested four times over an interval of 25-30,000 miles. As is evident from a comparison of Figs. 18 and 19, the two vehicle's emissions exhibit different mutagenic responses both in magnitude and in cycle-to-cycle differences. The values for Car 2 and Car 3 are almost mutually exclusive of each other and cover a range with an approximately five-fold change. The two sets of data are similar in that in each the FTP shows the largest test-to-test variations, the IDLE generally shows the lowest mutagenic activity and the overall activity decreases with accumulated mileage (higher test number in each case except for Test 30). Except for the lower IDLE values there are no apparent cycle-to-cycle differences. Fig. 20 shows data analogous to that in Figs. 18 and 19 but for an Oldsmobile 98 (Test 5), a Volkswagon Rabbit (Test 3), a Mercedes

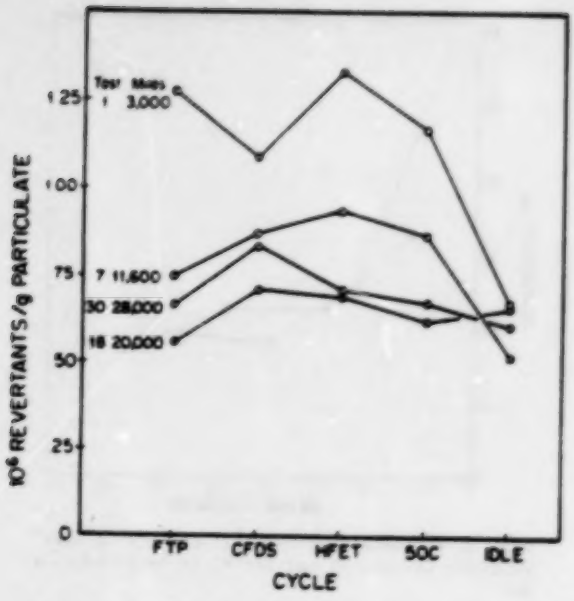

Fig. 18 - Ames Activity for Car #2 by Test and Cycle in Revertants /g Particulate.

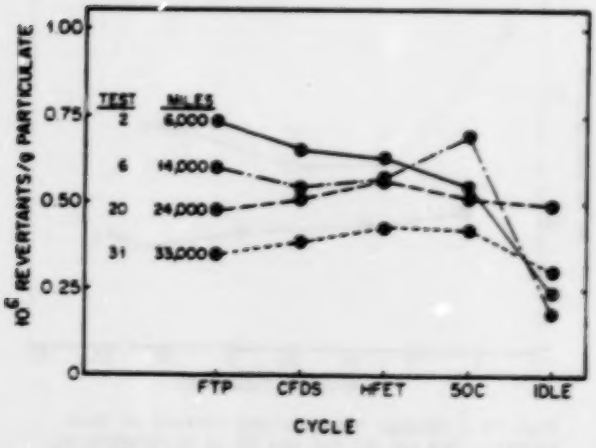

Fig. 19 - Ames Activity for Car #3 by Test and Cycle in Revertants /g Particulate.

Benz 300 CD (Test 15) and a Cadillac Eldorado (Test 9). These data are generally in the range of values exhibited the Oldsmobile Cutlass cruisers. These vehicles also display a wide divergence of absolute values and cycle-to-cycle variations.

Figs. 21, 22 and 23 display the data for each test in terms of revertants/mi and driving cycle. In this representation the test-to-test variations for a single vehicle are relatively small and cycle-to-cycle trends are more pronounced and reproducible. In each case, the number of revertants decreases in the order FTP>CFDS>HFET>50C (Test 8 on a Volkswagen has HFET slightly greater than CFDS); and the FTP shows the largest test-to-test differences. Except for Test 30, there is generally a reduction in revertants/mi as the vehicle

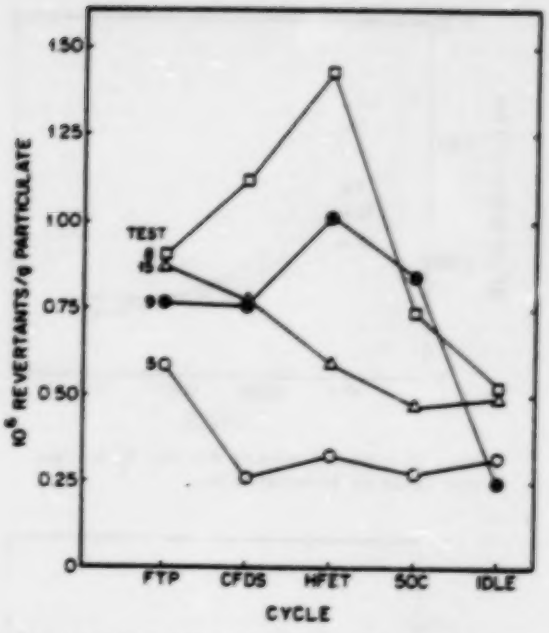

Fig. 20 - Ames Activity for Car #6, Car #13, Car #7 and Car #4 by Cycle in Revertants /g Particulate.

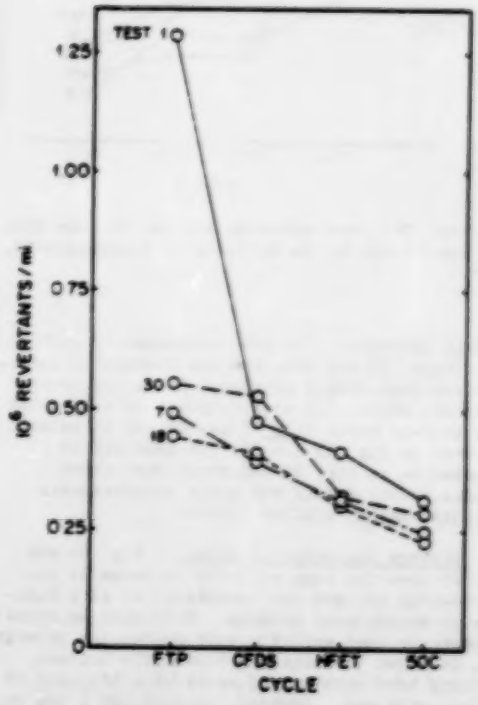

Fig. 21 - Ames Activity for Car #2 by Test and Cycle in Revertants/mi.

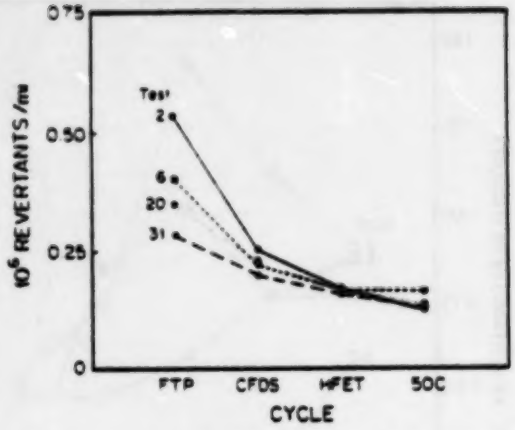

Fig. 22 - Ames Activity for Car #3 by Test and Cycle in Revertants/mi.

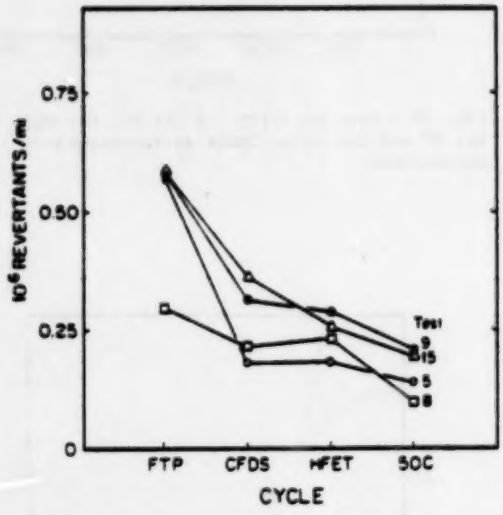

Fig. 23 - Ames Activity for Car #6, Car #13, Car #7 and Car #4 by Cycle in Revertants/mi.

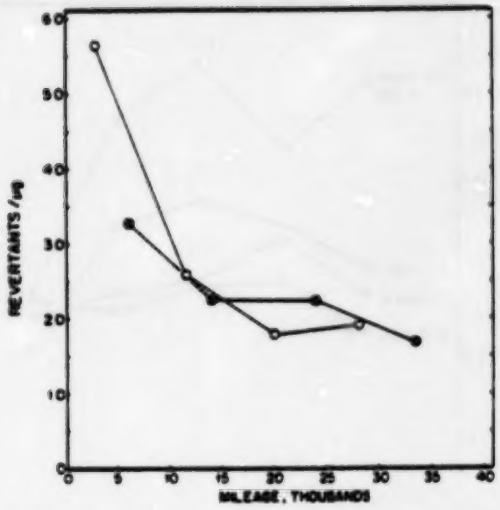

Fig. 24 - Mileage Accumulation Effects on Ames Activity for Car #2 and Car #3 in Revertants/µg SOF.

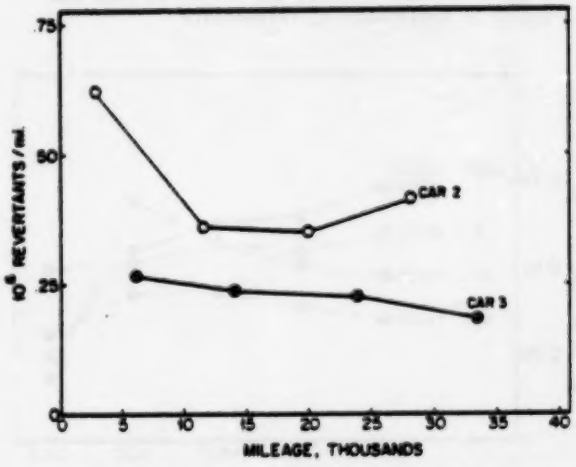

Fig. 25 - Mileage Accumulation Effects on Ames Activity for Car #2 and Car #3 in Revertants/mi.

mileage increases. As with revertants/g particulate data (Figs. 18 and 19), the two Oldsmobile Cutlass cruisers have widely divergent values of revertants/mi, especially when compared to each other on a mileage basis (c.f. Figs. 24 and 25 below). The data in Fig. 23 are for the same set of vehicles as in Fig. 20 and shows that other General Motors models and other manufacturers vehicles exhibit similar trends.

Mileage Accumulation Effect - Fig. 24 and Fig. 25 show the Ames activity in terms of revertants/ug extract and revertants/mi as a function of accumulated mileage. Both show an overall decrease in Ames activity with increasing mileage. Fig. 24 shows that the two Oldsmobile Cutlass cruisers have extracts of comparable activity at similar mileages. However, because Car 2 has both higher particulate emissions and % SOF it has considerably higher revertants/mi. Figures for revertants/Kg fuel and revertants/g particulate vs. mileage are essentially the same as Fig. 25 and, therefore, have not been included graphically but the tabular data are given in Appendix C. The large divergence of the lowest mileage point for Car 2 is possibly related to the fact that the vehicle had only 3,000 of usage at the time of the test.

Effect of Vehicle Test Phase - Table 9 contains average values and standard deviations for the vehicle test-phase ratios of specific activity (revertants/ug SOF) using the results from test-phase III as the comparison base. The sample number is considerably smaller than for similar comparisons presented above for particulate and SOF emissions, and combined with the inherent

Table 9-Variability of Ames Response by Vehicle Test Phase

Specific Activity (revertants/ug SOF)
TA98(-)

$$R = \left[\frac{\text{Phase I or II SOF activity (rev/ug)}}{\text{Phase III SOF activity (rev/ug)}}\right]$$

$\bar{R}$ = average of all R values from individual vehicle tests

		FTP	CFDS	HFET	30c	Idle
[Test Phase I / Test Phase III]	$\bar{R}$	1.16	1.14	1.17	0.98	1.14
	σ	0.80	0.59	0.28	0.28	0.43
	N	12	12	12	12	12
[Test Phase II / Test Phase III]	$\bar{R}$	1.31	1.14	1.24	0.96	1.01
	σ	0.50	0.30	0.31	0.23	0.33
	N	11	11	11	11	11

variability in the assay the average values presented must be considered as tentative. The standard deviations are large for all driving cycles, but the FTP appears to be significantly larger than other cycles. Ratios of activity between phases are fairly uniform for the driving cycle, and would suggest a 0-20% increase in specific activity for both phases I and II relative to phase III results, but the high standard deviation and limited sample size make this a preliminary estimate. Since phase III involves a fresh engine oil test condition whereas phases I and II are only a fuel switch, the uniformity of the results for comparisons of I/III and II/III are not completely surprising. The oil change effect on specific activity of SOF as measured by TA98(-) appears in these results not to be a massive effect.

CONCLUSIONS

The project to obtain emissions data from in-use diesel cars is not complete, and this paper is somewhat of a status report midway through the work. Conclusions at this point are preliminary and contingent upon data collection of vehicular emission factors over much larger spans of mileage accumulation, the inclusion of more engine configurations in the vehicle sample group, and a more thorough understanding of the chemistry of diesel particulate emissions as related to emission products, sampling procedures, and bioactivity.

Summarizing the data presented here, the following conclusions are drawn:

(1) Particulate Emissions Variability - Duplicate vehicle tests within a given fuel/oil condition gave a coefficient of variation of approximately 10% of the mean emission. Little differences were observed among vehicle makes or driving cycles tested.

The coefficient of variation for particulate emissions between driving cycles repeated at different fuel/oil conditions was approximately 10-15% of the mean emission rate, with little driving cycle or vehicle make dependence.

The coefficient of variation for SOF emissions (g/mi) between driving cycles repeated at different fuel/oil conditions was approximately 25% of the mean emission rate with little driving cycle or vehicle make dependence.

On the average, no systematic variations in either particulate or SOF emissions were found for the fuel/oil conditions tested although individual vehicle tests did exhibit significant changes, especially in terms of SOF. A more detailed analysis of the collected data (e.g. 5, 25) may reveal correlating parameters not accessed by overall averaging.

(2) Particulate Emissions - Particulate emissions (g/mi) generally were found to increase by driving cycle in the following order: 50c < HFET<CFDS<FTP. For the vehicles tested, particulate emissions increased in the order Mercedes-Benz < Volkswagon < General Motors.

Mileage accumulation effects for two General Motors vehicles tested up to 30,000 mi exhibited marked increases in particulate and SOF emissions up to 20,000 mi, and have subsequently stabilized for an undetermined period. Preliminary mileage accumulation effects on particulate for sample group vehicles with multiple tests show approximately 0.06 g/mi increase per 10,000 miles.

In general, particulate emissions expressed in time-based units increased in the following order: IDLE<FTP<50c<CFDS<HFET, and Volkswagon < Mercedes-Benz < General Motors.

Particulate emissions expressed on a time basis show little variation among driving cycles for General Motors vehicles, with the exception of the idle mode where emissions averaged 60% of the average for driven cycles. Particulate emissions in the idle mode for Volkswagon and Mercedes-Benz vehicles averaged 10-20% of the time-based emissions from driven cycles.

Particulate emissions of Volkswagon and Mercedes-Benz vehicles exhibited a stronger driving cycle dependence when expressed in (g/min) than as (g/mi).

(3) Fuel Specific Emissions - Fuel specific particulate emissions increased in the order: HFET<CFDS<FTP for General Motors vehicles, but exhibited no definite trend for Volkswagon or Mercedes-Benz types, and increase in the order Mercedes-Benz < General Motors < Volkswagon.

Fuel specific particulate emission averages were in the range 3.5-5.5 g/Kg fuel consumed for all vehicles and FTP, CFDS, and HFET driving cycles.

Fuel specific SOF emissions averages were in the range 0.5-1.2 g SOF/Kg fuel consumed for all vehicles tested on the FTP, CFDS and HFET driving cycles. Fuel specific SOF (g/mi) showed no definitive driving cycle dependence but increased in the order Mercedes-Benz<Volkswagon<General Motors.

(4) SOF Emissions - Average SOF expressed as a percentage of collected particulate increased in the order IDLE = FTP<CFDS<HFET<50c for General Motors vehicles. SOF (%) was relatively constant

for all cycles for Mercedes-Benz vehicles at 13%, and averaged about 20% for Volkswagon, except in the idle mode where Volkswagon SOF (%) averaged 60%.

Mass emission of SOF (g/mi) increased in the general order 50c<HFET<CFDS<FTP, and Mercedes-Benz < Volkswagon<General Motors.

(5) Ames Test Activity of SOF Samples - All SOF samples which have been tested exhibit a positive mutagenic response in the Ames test. One set of SOF samples kept in cold storage and assayed at a 5-month interval showed an average 25% reduction in specific activity.

For multiple tests on two General Motors vehicles, mutagenic activity of SOF samples exhibited a general decrease with accumulated mileage (0-30,000 mi) whether expressed as activity/gram of particulate or activity of SOF emissions/mi.

No definitive effect of driving cycle on mutagenic activity was identified, except that SOF samples from the idle mode frequently showed a decreased response.

At this time it is not possible to partition the observed mutagenic activity of SOF samples into emission products and sampling artifacts. The decreased activity noted at idle could be related to engine variables or exhaust dilution variations, for example.

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Appendix A

AEL Reference Fuel Specification Data

Distillation

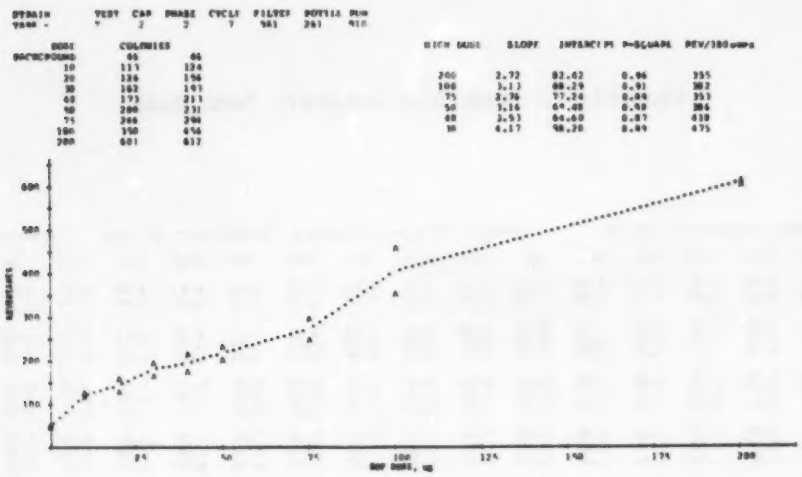
Initial Boiling Point	336°F
10% Recovery	398°F
20% "	423°F
30% "	442°F
40% "	461°F
50% "	483°F
60% "	501°F
70% "	522°F
80% "	546°F
90% "	585°F
95% "	600°F
End Point	635°F
% Recovery	98.8
% Residue	1.1
% Loss	0.1

Elemental Analyses

Carbon	86.33%
Hydrogen	13.06%
Nitrogen	<0.05%
Oxygen	0.47%
Sulfur	0.14%

Ash	<0.001%
API Gravity @ 60°F	36.2
Specific Gravity @ 60°F	0.8438
Heating valve (BTU/lb)	19,458
Cetane Index	45.5
% Saturates	67.1
% Olefins	0.3
% Aromatics	32.6

Appendix B-Typical Dose Response Curves for TA98(-) Ames Tests



Appendix C- Ames Test Activity Data Base

			Slope (revertants / gm SOF)					Revertants*/ gram Particulate					Revertants*/ MJ fuel			Revertants*/ Mile			
TEST	CAR	PHASE	FTP	CFDS	HFET	SOC	IDLE	FTP	CFDS	HFET	SOC	IDLE	FTP	CFDS	HFET	FTP	CFDS	HFET	SOC
1	2	1	8.00	4.40	5.90	4.10	5.00	1.63	1.09	1.57	0.83	0.87	8.54	4.47	4.94	1.32	0.51	0.51	0.27
1	2	3	10.40	5.40	5.00	5.20	2.90	1.76	1.07	1.09	1.49	0.47	9.31	3.74	2.84	1.14	0.44	0.30	0.34
2	3	1	5.20	3.90	3.60	2.20	2.40	0.74	0.70	0.71	0.50	0.24	3.77	3.37	1.85	0.56	0.28	0.20	0.12
2	3	2	5.00	2.80	2.90	2.00	2.10	0.74	0.51	0.62	0.53	0.23	3.51	2.46	1.51	0.51	0.19	0.16	0.12
2	3	3	5.20	4.70	2.80	2.20	2.00	0.72	0.75	0.55	0.58	0.23	3.41	3.35	1.22	0.53	0.28	0.14	0.12
5	4	1	4.10	2.20	1.70	1.50	1.20	0.59	0.42	0.36	0.34	0.28	4.06	2.49	1.90	0.59	0.29	0.20	0.17
5	4	2	3.30	0.80	1.60	0.70	1.40	0.75	0.15	0.36	0.16	0.31	4.98	0.84	1.79	0.72	0.10	0.19	0.08
5	4	3	2.50	0.80	1.00	1.20	1.50	0.41	0.20	0.24	0.30	0.34	2.78	1.18	1.41	0.41	0.14	0.15	0.16
6	3	1	5.60	2.00	2.20	2.20	1.40	0.85	0.46	0.57	0.67	0.18	4.06	1.58	1.52	0.59	0.19	0.16	0.16
6	3	2	3.60	2.50	2.60	2.10	1.60	0.59	0.58	0.68	0.62	0.19	2.73	1.96	1.84	0.39	0.24	0.20	0.14
6	3	3	1.60	1.90	1.40	1.90	1.00	0.36	0.57	0.43	0.77	0.15	1.58	1.99	1.24	0.22	0.24	0.13	0.20
7	2	1	3.20	2.30	2.60	1.90	2.50	0.66	0.76	0.96	0.88	0.52	2.78	2.98	3.01	0.41	0.33	0.32	0.23
7	2	2	4.30	3.80	2.90	1.80	2.70	0.84	0.90	1.02	0.84	0.54	3.96	3.39	3.10	0.56	0.40	0.32	0.24
7	2	3	2.90	2.60	2.00	1.80	2.20	0.74	0.95	0.81	0.88	0.49	3.37	3.75	2.48	0.50	0.44	0.29	0.25
8	6	1	4.20	4.00	4.40	3.10	1.40	0.93	1.03	1.17	0.59	0.51	3.50	3.45	3.44	0.24	0.21	0.21	0.09
8	6	2	4.80	5.20	5.90	4.00	1.20	0.89	1.15	1.30	0.72	0.45	3.08	4.06	3.87	0.22	0.25	0.21	0.10
8	6	3	3.30	3.90	4.80	3.70	1.10	0.86	1.14	1.71	0.87	0.58	6.17	3.39	5.49	0.43	0.19	0.28	0.11
9	7	1	2.40	2.10	3.20	1.70	1.20	0.55	0.63	0.99	0.78	0.22	3.64	2.10	2.75	0.57	0.25	0.28	0.21
9	7	2	4.00	2.70	3.00	2.60	1.60	0.85	0.80	0.87	0.96	0.26	4.14	2.63	2.45	0.64	0.32	0.25	0.25
9	7	3	3.70	3.00	3.10	2.20	1.40	0.88	0.83	1.17	0.77	0.25				0.98	0.39	0.34	0.19
15	13	1	7.70	7.40	8.90	5.70	7.80	0.80	0.68	0.72	0.58	0.75	4.80	2.82	2.82	0.54	0.31	0.29	0.21
15	13	2	10.40	10.70	7.00	4.60	3.10	0.97	0.95	0.55	0.40	0.38	4.59	3.91	2.31	0.62	0.45	0.25	0.19
15	13	3	9.40	8.40	7.10	5.20	4.80	0.83	0.67	0.49	0.42	0.42	3.94	2.64	1.85	0.54	0.30	0.21	0.16
18	2	1	1.30	1.10	1.10	1.10	2.50	0.44	0.47	0.54	0.57	0.37	2.34	2.25	3.09	0.35	0.27	0.23	0.21
18	2	2	1.80	1.80	1.80	1.60	2.70	0.54	0.77	0.82	0.57	0.66	2.99	4.02	3.38	0.44	0.48	0.37	0.22
18	2	3	2.30	2.20	1.40	1.30	2.90	0.65	0.85	0.68	0.67	0.70	3.74	4.10	2.74	0.55	0.49	0.30	0.25
20	3	1	1.90	1.90	1.60	0.81	1.70	0.37	0.47	0.45	0.29	0.26	1.92	1.92	1.38	0.27	0.22	0.14	0.08
20	3	2	3.00	2.30	1.90	1.90	2.40	0.59	0.63	0.58	0.46	0.33	3.19	2.62	1.70	0.44	0.29	0.18	0.10
20	3	3	2.20	1.50	2.00	1.90	6.70	0.46	0.41	0.65	0.58	0.89	2.47	1.74	1.98	0.35	0.20	0.21	0.16
30	2	1	2.20	2.20	1.40	1.10	2.00	0.60	0.94	0.66	0.59	0.52	3.21	5.09	2.68	0.48	0.58	0.29	0.22
30	2	2	2.30	2.30	1.90	1.50	2.80	0.64	0.96	0.91	0.78	0.75	3.48	5.31	3.58	0.51	0.62	0.39	0.32
30	2	3	2.20	1.50	1.40	1.30	2.20	0.73	0.61	0.57	0.66	0.56	4.29	3.10	2.17	0.65	0.36	0.24	0.29
31	3	1	1.40	1.80	1.60	1.60	3.60	0.25	0.45	0.48	0.63	0.38	1.37	2.06	1.59	0.19	0.23	0.17	0.19
31	3	2	1.70	1.40	1.20	0.60	2.00	0.30	0.35	0.38	0.21	0.24	1.89	1.54	1.16	0.27	0.17	0.13	0.06
31	3	3	2.30	1.20	1.30	1.00	2.20	0.46	0.32	0.41	0.39	0.25	2.55	1.45	1.35	0.37	0.16	0.14	0.11

*In units of 10^6

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